

Evaluating Dentin Surface Treatments for Resin-Modified Glass Ionomer Restorative Materials

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Clinical Relevance

A dentin bonding agent provided greater bond strength to dentin for RMGI than the manufacturers' recommended products.

SUMMARY

This *in vitro* study evaluated the effect of six surface treatments on the shear bond strength of three resin-modified glass ionomers (RMGIs) to dentin. Occlusal surfaces of caries-free third molars were reduced to expose only dentin. Surface treatments were smear

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DOI: 10.2341/12-162-L

layer intact (negative control), Cavity Conditioner, EDTA, Ketac Primer, Self Conditioner, and etching with 35% phosphoric acid followed by the application of Optibond Solo Plus. Filtek Z250 composite resin bonded with Optibond Solo Plus served as a positive control. Conditioning agents were used according to the manufacturers' instructions. After surface treatments, Fuji II LC, Riva LC, Ketac Nano, and Filtek Z250 were placed in copper-band matrices 5 mm in diameter and 2 mm in height and were light-cured for 20 seconds. Specimens were stored in 100% humidity for 24 hours, after which they were placed in deionized water for 24 hours at 37°C. They were then tested under shear forces in an Instron Universal Testing Machine at a crosshead speed of 0.5 mm/min. A two-way analysis of variance and Tukey honestly significant difference statistical analyses ($p < 0.05$) indicated significant interaction between RMGIs and conditioning agents. Acid etching followed by Optibond Solo Plus provided highest bond strengths for all three RMGIs, which were not statistically different from the positive control.

INTRODUCTION

Resin-modified glass ionomers (RMGIs) are the result of grafting water-soluble monomers such as 2-hydroxyethyl methacrylate (HEMA) to polyalkenoic acid chains of conventional glass ionomers (CGIs) and incorporating resins and photoinitiators.^{1,2} The fluoride release of RMGIs is comparable and often better than that of CGIs.²⁻⁵ Advantages of RMGIs compared with CGIs include increased working time, decreased setting time, ease of handling, and improved physical properties and esthetics.¹⁻⁴ Resin-modified glass ionomers have been marketed as liners, bases, dentin bonding agents, sealants, luting agents, and core foundations, as well as direct restorative materials, particularly for class V lesions.¹⁻³

There are two distinct bonding mechanisms for RMGIs to tooth structure: a chemical bonding between anions of polyalkenoic acid chains and calcium ions in hydroxyapatite¹⁻⁴ and a micromechanical bond similar to that which occurs between dentin bonding agents and dentin.⁶⁻¹⁰ Several agents have been evaluated to condition dentin prior to application of CGIs and RMGIs. These have included citric, polyacrylic, phosphoric, tannic, and ethylenediaminetetraacetic (EDTA) acids.¹¹⁻¹³ Polyacrylic acid has been the mainstay for conditioning dentin prior to application of CGIs and RMGIs. Recently, manufacturers have recommended other conditioners to replace polyacrylic acid; they include Ketac Primer (3M ESPE, St Paul MN, USA) and Self Conditioner (GC, Tokyo, Japan).

Resin-modified glass ionomers are packaged in capsules as a powder and liquid or as two separate pastes. Examples of powder/liquid RMGIs include Fuji II LC (GC) and Riva LC (Southern Dental Industries, Victoria, Australia), which both require trituration. Ketac Nano (3M ESPE) contains two separate pastes that are mixed together when dispensed through an auto-mixing tip. In lieu of using polyacrylic acid to condition dentin, the manufacturer of Ketac Nano recommends conditioning with Ketac Primer. When using Fuji II LC, the manufacturer recommends dentin conditioning with either Cavity Conditioner (GC) or Self Conditioner. Cavity Conditioner contains 20% polyacrylic acid and 3% aluminum chloride. Self Conditioner contains HEMA and 4-methacryloxyethyl trimellitate anhydride (4-META) and is purported by the manufacturer to provide stronger and more stable bonding. In addition to containing hydrophilic resins, both Ketac Primer and Self Conditioner are acidic, with a pH of 3.0 and 1.8, respectively.

Southern Dental Industries, manufacturer of Riva LC, recommends using either polyacrylic or phosphoric acid for dentin conditioning. Because RMGIs contain resin, it has been hypothesized that the bond strength of RMGIs can be improved by the application of dentin bonding agents. The purpose of this *in vitro* study was to compare the effect of five different conditioning protocols (polyacrylic acid, EDTA, Ketac Primer, Self Conditioner, and a dentin bonding agent) on the shear bond strength of three different RMGIs to dentin. In addition, a negative control was included that consisted of leaving the smear layer intact. A group consisting of Filtek Z250 composite resin (3M ESPE) bonded to dentin with Optibond Solo Plus (Kerr, Orange, CA, USA) after phosphoric acid etching was used as a positive control. The null hypothesis we tested was that there would be no difference in bond strengths as a result of surface treatments or RMGIs.

METHODS AND MATERIALS

Dental materials and their compositions are listed in Table 1. Recently extracted, caries-free third molars were cleaned of debris and disinfected in a 0.5% solution of sodium hypochlorite and sterile water for 30 minutes. Teeth were embedded in phenolic rings (Buehler Ltd, Lake Bluff, IL, USA) 2 mm apical to their cemento-enamel junctions using Orthodontic Resin (LD Caulk/Dentsply, Milford, DE, USA). The occlusal surface of each tooth was carefully reduced using a conventional model trimmer (Handler Mfg Co, Garwood, NJ, USA) with water to produce a dentin surface. The surface was not treated with any other abrasive papers or discs. Conditioners, primers, and bonding agent were applied by one investigator according to the manufacturers' instructions as described in Table 2. Copper-band matrices 5 mm in diameter and 2 mm in height were held on the dentin surface by a second investigator by grasping the copper band with a cotton forceps and holding the band steady against the flat dentin surface. The first investigator activated, triturated, or mixed the RMGI according to the manufacturer's instructions. The first investigator placed the RMGI or Z250 resin into the copper band, placed a polyester strip on the coronal aspect of the copper band, and used finger pressure to compress the restorative materials against dentin. Using a SmartLite IQ (LD Caulk/Dentsply) with an intensity of 500 mW/cm², the first investigator photo-cured the RMGI or Z250 for 20 seconds while the second investigator held the copper band steady. Fifteen specimens were made for each experimental and control group (see Figure

Table 1: <i>Composition of Materials</i>	
Material	Composition
RMGI	
Fuji II LC	Powder
	Fluoro-alumino-silicate glass
	Liquid
	Polyacrylic acid, HEMA, trimethylene dicarbonate, and other proprietary ingredients
Ketac Nano LC	Paste A
	Silanted glass, zirconia and silica HEMA, PEGDMA, Bis-GMA, TEGMA, fluoro-alumino-silicate glass
	Paste B
	Silanated ceramic, water, HEMA, and copolymer of acrylic and itaconic acids
Riva LC	Liquid
	Polyacrylic and tartaric acids, HEMA, DMA, and acidic monomer
	Powder
	Fluoro-alumino-silicate glass and polyacrylic acid
Composite	
Filtek Z250	Silane-treated ceramic filler, bisphenol A polyethylene glycol diether DMA, diurethane DMA, Bis-GMA, TEGDMA
Conditioning Agents	
EDTA	17% ethylene-diamine-tetracetic acid
Cavity Conditioner	20% polyacrylic acid and 3% aluminum chloride
Self Conditioner	HEMA, 4-META, ethanol, and water
Ketac Primer	HEMA, water, and copolymer of acrylic and itaconic acids
Optibond Solo Plus	Bisphenol glycidyl methacrylate, glycerol DMA, glycerol phosphate DMA, DMAs, ethanol silicone oxide, barium borosilicate, and sodium hexafluorosilicate ₆
<i>Abbreviations: Bis-GMA, bisphenol A glycidyl methacrylate; DMA, dimethacrylate; 4-META, 4- methacryloxyethyltrimellitate anhydride; HEMA, 2-hydroxyethyl methacrylate; PEGDMA, polyethylene glycol dimethacrylate; TEGDMA, triethylene glycol dimethacrylate.</i>	

Table 2: <i>Conditioning Protocols</i>	
Agent	Manufacturer Instructions
Cavity Conditioner	1. Apply for 10 seconds.
	2. Rinse thoroughly with air/water aerosol and gently dry with air but do not desiccate.
EDTA	1. Apply for 60 seconds.
	2. Rinse thoroughly with air/water aerosol and gently dry with air but do not desiccate.
Self Conditioner	1. Apply a thin layer on dentin and leave undisturbed for 10 seconds.
	2. Dry for five seconds with air but do not desiccate.
Ketac Primer	1. Apply for 15 seconds, keeping the surface wet with primer.
	2. Gently dry with air for 10 seconds.
	3. Photo-cure for 10 seconds.
Optibond Solo Plus	1. Apply 37% phosphoric acid for 10 seconds and thoroughly rinse with an air/water aerosol for 10 seconds.
	2. Apply bonding agent with a scrubbing motion for 10 seconds, and evaporate the solvent for 10 seconds using air.
	3. Repeat Step 2 and photo-cure for 20 seconds.

1). Excess restorative material was removed from the matrix/dentin interface with a sharp #25 Bard Parker blade (Miltex, York, PA, USA). The samples were stored for a total of 48 hours prior to testing. Initially, they were stored for 24 hours in 100% humidity at 37°C. They were then removed from the humidor and placed in 37°C deionized water for 24 hours. Samples were placed in an Instron Universal Testing Machine (Model TTC, Instron Corporation, Canton, MA, USA) and shear tested with a cross-head speed of 0.5 mm/min. A shearing bar beveled to a 1-mm-thick contact surface area was placed at the junction of dentin and copper band matrix (see Figure 2). The load required for failure was recorded in pounds and converted to megapascals (MPa). To assess the effect of the independent variables

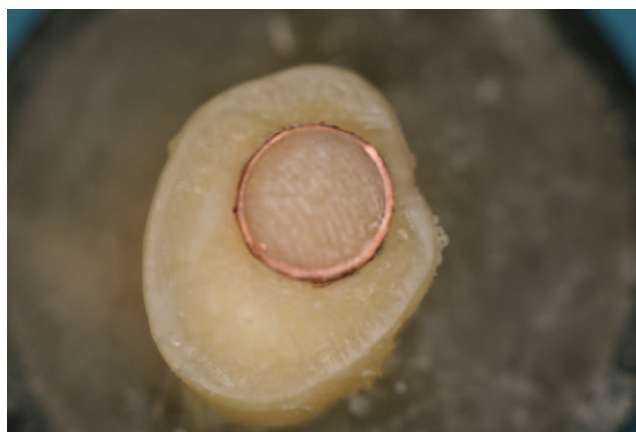


Figure 1. *Completed specimen.*

(surface treatment and RMGI), the parametric data were analyzed with a two-way analysis of variance (ANOVA). A Tukey honestly significant difference multiple comparison procedure was used to assess significant differences among groups at a confidence level of $p < 0.05$. All analyses were performed using SAS software (SAS Institute Inc, Cary NC, USA). After testing, the specimens were visually inspected with 4.8× magnification to classify failures as cohesive, adhesive, or mixed.

RESULTS

The minimum, maximum, mean shear bond strengths, standard deviations, and failure modes are listed in Table 3. The data were skewed and therefore analyzed on a log-transformed scale. A two-way ANOVA indicated significant differences between groups ($p < 0.0001$) and significant interactions between RMGIs and conditioning agents. The results for each RMGI and conditioning agent will be presented and discussed individually (see Figure 3).

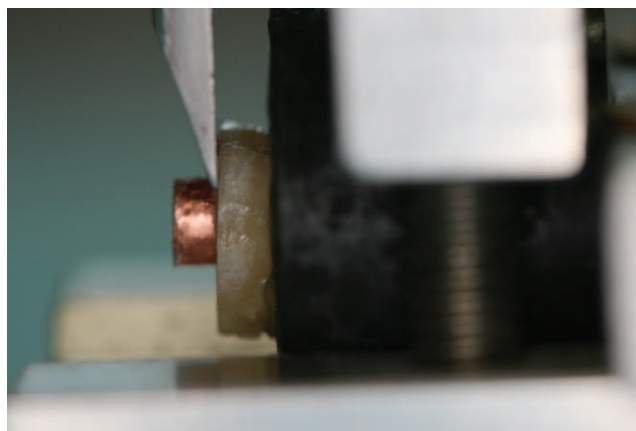


Figure 2. *Specimen in the Instron.*

Table 3: Shear Bond Strength (MPa)					
RMGI and Conditioning Agent	Min	Max	Mean	SD	Failure Mode (Adhesive, Cohesive, Mixed)
Fuji II LC, Smear Layer	1.85	6.50	3.47	1.18	(15, 0, 0)
Fuji II LC, Cavity Conditioner ^a	1.81	8.79	4.99 ^{DEF^b}	2.07	(15, 0, 0)
Fuji II LC, EDTA	5.14	10.68	7.90 ^{BCD}	1.75	(15, 0, 0)
Fuji II LC, Ketac Primer	6.60	12.20	9.36 ^{ABC}	1.74	(15, 0, 0)
Fuji II LC, Self Conditioner	7.24	12.20	9.37 ^{ABC}	1.49	(15, 0, 0)
Fuji II LC, OBSP	10.13	15.13	12.11 ^{AB}	1.50	(12, 0, 3)
Ketac Nano, Smear Layer	1.00	3.88	1.79	0.83	(15, 0, 0)
Ketac Nano, Cavity Conditioner	0.27	7.80	1.70 ^G	2.68	(15, 0, 0)
Ketac Nano, EDTA	2.00	9.90	4.76 ^{DEF}	2.73	(15, 0, 0)
Ketac Nano, Ketac Primer ^a	1.90	6.78	3.72 ^{EF}	1.44	(15, 0, 0)
Ketac Nano, Self Conditioner	5.10	10.32	7.66 ^{BCD}	1.61	(15, 0, 0)
Ketac Nano, OBSP	8.70	16.60	12.11 ^{AB}	2.23	(13, 0, 2)
Riva LC, Smear Layer	2.88	8.23	4.76	1.56	(15, 0, 0)
Riva LC, Cavity Conditioner ^a	1.70	7.08	3.63 ^F	1.56	(15, 0, 0)
Riva LC, EDTA	2.37	6.82	3.86 ^{EF}	1.56	(15, 0, 0)
Riva LC, Ketac Primer	2.40	6.40	3.59 ^F	1.20	(15, 0, 0)
Riva LC, Self Conditioner	4.89	7.08	6.15 ^{CDE}	0.75	(15, 0, 0)
Riva LC, OBSP	13.30	18.70	15.47 ^A	1.75	(12, 0, 3)
Filtek Z-250, OBSP	10.88	19.73	14.63 ^A	2.88	(12, 0, 3)

Abbreviations: Max, maximum; Min, minimum; OBSP, Optibond Solo Plus; RMGI, resin-modified glass ionomer; SD, standard deviations.
^a Indicates the manufacturer's recommended conditioner.
^b Tukey multiple comparison procedure: Means not sharing the same letter are significantly different ($p < 0.05$).

For Fuji II LC the five conditioning protocols were significantly different and higher than leaving the smear layer intact ($p < 0.013$). Regarding Ketac Nano, all conditioning agents were significantly different and higher than the negative control except for Cavity Conditioner ($p = 0.7335$). When bonding

Riva LC, none of the conditioning agents were stronger than the negative control ($p < 0.05$) except Optibond Solo Plus.

Comparing the three RMGIs within each conditioning group, all RMGIs were significantly different from one another when the smear layer was

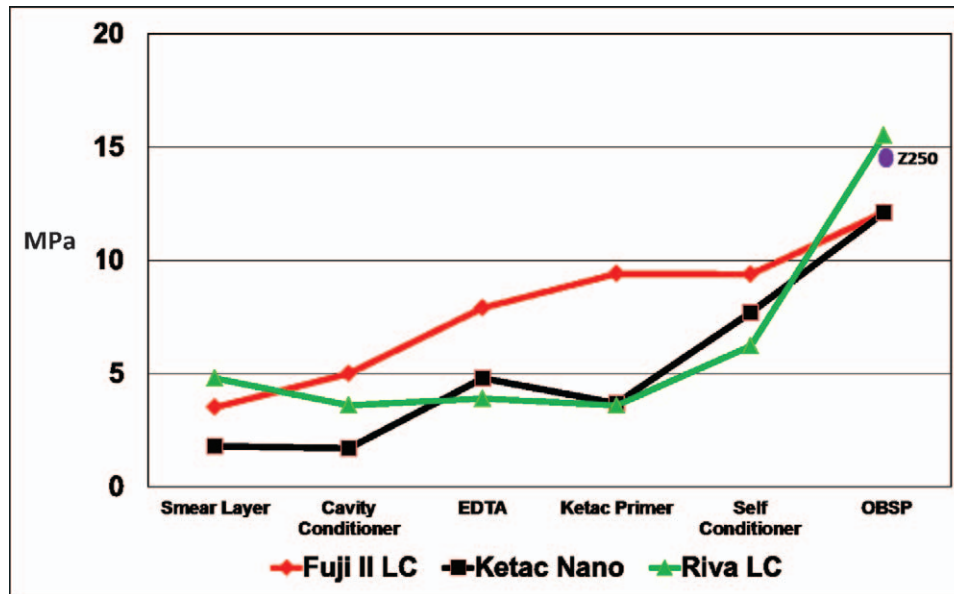


Figure 3. Graph of the shear bond strengths of three RMGIs.

intact ($p < 0.031$), with Riva LC (4.8 MPa) significantly stronger than the others. Additionally, all three RMGIs were significantly different from one another when Cavity Conditioner was used ($p < 0.0297$), with Fuji II LC (5.0 MPa) stronger than the others. When conditioning with EDTA, Ketac Nano (4.8 MPa) and Riva LC (3.9 MPa) were not different ($p > 0.15$), but they were weaker than Fuji II LC (7.9 MPa) ($p < 0.0006$). Within the Ketac Primer group, Fuji II LC (9.4 MPa) was significantly stronger than Ketac Nano (3.7 MPa) and Riva LC (3.6 MPa) ($p < 0.031$), which were not significantly different from each other ($p > 0.08$). When Self Conditioner was used, Fuji II LC (9.4 MPa) and Ketac Nano (7.7 MPa) were not different from each other ($p > 0.13$), but both were stronger than Riva

LC (6.2 MPa) ($p = 0.0042$). There was no difference among the three RMGIs and Z250 when Optibond Solo Plus was used ($p = 0.2083$).

DISCUSSION

The null hypothesis that surface treatments would not have an effect on the shear bond strength of three RMGIs to dentin was rejected. Using visual examination of the fractured specimens, we observed predominately adhesive failures for all groups except those that were bonded with Optibond Solo Plus. In these groups, including the positive control, two to three specimens from each group exhibited a mixed failure in which the dentin and RMGI fractured (see Figure 4). This would suggest these bonding configurations surpassed the inherent strength of the RMGI and dentin, or dentin and the RMGI may have been weakened by the presence of structural flaws. Therefore, the true maximum bond strength could not be measured when dentin or RMGI fractured during testing.

The overall question of which combination is best may be answered by comparing all of the experimental groups excluding the negative control (see Table 3). The Tukey multiple comparison procedure indicated that all three RMGIs when used with a total etch technique and Optibond Solo Plus were statistically equal to the positive control or the resin-dentin bond strength. Additionally, Fuji II LC used with Ketac Primer or Self Conditioner was equal to the positive control ($p > 0.05$).

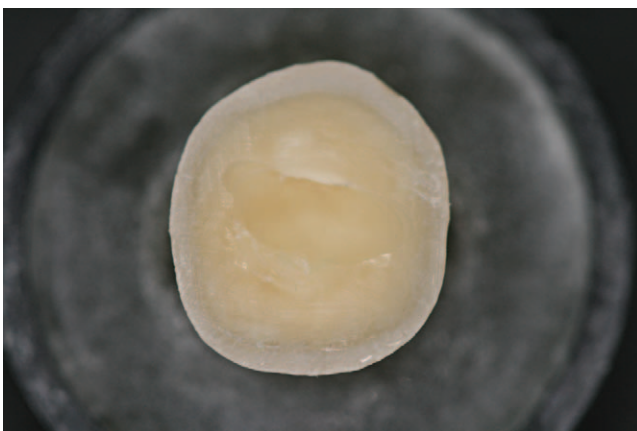


Figure 4. Mixed failure in which dentin and RMGI fractured.

The shear bond strength of the Fuji II LC with the smear layer intact (3.5 MPa) is nearly the same as that in a study by Hajizadeh and colleagues,¹⁴ who reported shear bond strength of 3.1 MPa. There are several explanations why RMGIs are able to bond to the smear layer. First, RMGIs contain polyacrylic acid (polyalkenoic acid chains), which acts as a mild self-conditioner.¹⁵ Second, the smear layer contains calcium ions that may provide bonding sites for chemical bonding with the polyalkenoic acid chains in the RMGI. Furthermore, the inherent dentin irregularities produce during specimen preparation provided micromechanical retention.

The purpose of Cavity Conditioner (polyacrylic acid) is to remove the smear layer without completely unplugging the dentin tubules. The exposed calcium ions within hydroxyapatite are available for chemical bonding with the carboxyl groups of the polyalkenoic acid. Any collagen that becomes exposed may provide additional micromechanical retention. The addition of aluminum chloride in Cavity Conditioner is thought to stabilize the collagen matrix during demineralization, allowing better penetration of the RMGI.¹⁶ This study did not demonstrate any benefit of using Cavity Conditioner, which, ironically, two manufacturers recommend as the conditioning agent. This finding is supported by several other studies.¹⁶⁻²¹

Increased bond strengths with EDTA for Fuji II LC and Ketac Nano may be the result of enhanced micromechanical retention due to improved removal of the smear layer and dentinal plugs. Additionally, EDTA does not alter the fibrillar structure of collagen, allowing the mineral content of collagen to bond with the ionic component of RMGIs.²² Fagundes and others²³ demonstrated increased microtensile bond strength of Fuji II LC and Vitremer (3M ESPE) following pretreatment with EDTA compared with polyacrylic acid. Additionally, Nakanuma and others²⁴ using Fuji II LC demonstrated greater tensile bond strength when EDTA was combined with experimental dentin primers and a bonding agent than when used with polyacrylic acid.

The purpose of primers such as Ketac Primer and Self Conditioner is to improve the wettability of dentin, allowing improved monomer penetration into the hydrophilic dentin substrate. Primers contain unsaturated carbon-carbon bonds that may lead to direct covalent bonding with resin constituents of RMGIs when polymerized. If the pH of the primer is low enough, it may also partially remove the smear layer, allowing the RMGI matrix to penetrate into the

superficial layer of dentin, creating a cement-matrix-dentin interdiffusion zone.²⁵ Ketac Primer and Self Conditioner have a pH of 3.0 and 1.8, respectively. Self Conditioner, with a lower pH, may have more completely removed the smear layer than Ketac Primer, resulting in higher bond strengths for Ketac Nano and Riva LC. However, we did not observe the same effect with Fuji II LC because Ketac Primer (9.4 MPa) and Self Conditioner (9.4 MPa) produced the same bond strengths, suggesting that the RMGIs react differently with specific conditioners and primers. Other investigators support the fact that primers compared with polyacrylic acid increased bond strength of RMGI to dentin.^{18,25-27}

Riva LC performed best only when used with Optibond Solo Plus after total etching. This suggests that Riva LC behaves more like a resin than a CGI. In this study all three RMGIs obtained their highest bond strengths when Optibond Solo Plus was applied after etching dentin with phosphoric acid. Obtaining improved bond strengths with bonding agents compared with other conditioning agents is supported by other investigations.^{21,28,29} Micromechanical bonding through the hybrid layer for RMGIs has been demonstrated with different conditioners producing different degrees of demineralization and hybrid layer thickness.¹⁶ Dentin bonding agents are able to form a chemical union with RMGIs due to the presence of HEMA and other resins in RMGIs. Why manufacturers continue to recommend the use of polyacrylic acid as a conditioner for RMGIs despite the knowledge that they contain resin is not fully understood. Development of dentin conditioners for CGIs occurred in the late 1970s, a decade before acid etching dentin was an accepted procedure. Many of the conditioning agents evaluated removed the smear layer, opened dentin tubules, and were considered detrimental.¹⁷ The calcium-rich hydroxyapatite was thought to be unavailable for bonding with the polyalkenoic acid chains in conventional glass ionomers. The tradition of using polyacrylic acid was simply carried over for use with RMGIs.

Results of this study suggest the hybrid layer may be the major source of bond strength, whereas chemical bonding may play an important role in providing marginal integrity, bond durability, and extending the longevity of the restoration.^{30,31} In addition to increased bond strength, an advantage of using a dentin bonding agent is that the sandwich technique in which a resin is veneered over a RMGI is simplified by using a single conditioning step that works both for composites and RMGIs.

Clinicians may be reluctant to use dentin bonding agents with RMGIs because there may be a decrease in the amount of fluoride released. It has been demonstrated that a dentin bonding agent significantly reduced, but did not prevent, fluoride passage.^{32,33} However, the minimal quantity of fluoride necessary to clinically inhibit the caries process has never been established. Using silver nitrate ions and scanning electron microscopy, Sano and others³⁴ demonstrated leakage within the hybrid layer. Given that silver nitrate ions used in their study have similar dimensions as fluoride ions, one may postulate that fluoride ions may also penetrate through the hybrid layer and into adjacent dentin to provide cariostatic properties.³⁴

Setting reaction of RMGIs relies on two different mechanisms that depend and compete with each other.³⁵ Khoroushi and others³⁶ demonstrated that delaying light polymerization of Fuji II LC for two minutes, thus allowing the acid-base chemical reaction to proceed first, resulted in significantly decreased shear bond strength. Therefore, for maximum bond strength it is important to photo-cure RMGIs soon after placement. Any remaining RMGI not photo-cured may continue to undergo the acid-base setting reaction but at a reduced rate.

It is impossible to uniformly compare results across different studies. Slight differences in methodologies produce varying results. Some studies bond a column of RMGI; others bond the RMGI, then apply a column of composite resin on top of it. Resin-modified glass ionomers have been tested as dentin bonding agents, luting agents, liners/bases or restorative materials. It is unacceptable to uniformly compare results for all types of RMGI materials. Additionally, some studies have used human teeth and others bovine incisors. Research using microtensile and microshear bond methodologies have produced much greater bond strengths than what was reported in this study.^{37,38} When the bonding surface can be kept small (1 mm² for microtensile and microshear methodologies), there is a more homogeneous stress distribution. The bonding surface area in this study was 19 mm². When larger surface areas are used the bonding material will contain larger flaws and voids resulting in higher stress concentrations in these areas that lead to lower bond strengths. Additionally, a 1-mm flat-edge chisel used in this study causes greater stress concentration at the area of load application.³⁹ However, having the RMGI surrounded by a copper band may have allowed the load to be applied more evenly due to copper having a higher modulus than

RMGI.^{40,41} We believe this to be similar to using a wire loop or notched bar for load application.^{39,42} Promising laboratory results do not necessarily guarantee clinical success. Rather, *in vitro* studies serve as a good screening tool and guide for selection of dental materials and procedures that may be tested clinically.

CONCLUSIONS

1. The use of Cavity Conditioner did not significantly improve the bond strength of the three RMGIs compared with leaving the smear layer intact.
2. The effect of conditioning agent was dependent upon the RMGI.
3. All three RMGIs bonded with Optibond Solo Plus after acid etching produced shear bond strengths that were not statistically different than the composite-dentin bond.

Conflict of Interest

The authors of this article certify that they have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

(Accepted 21 August 2012)

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